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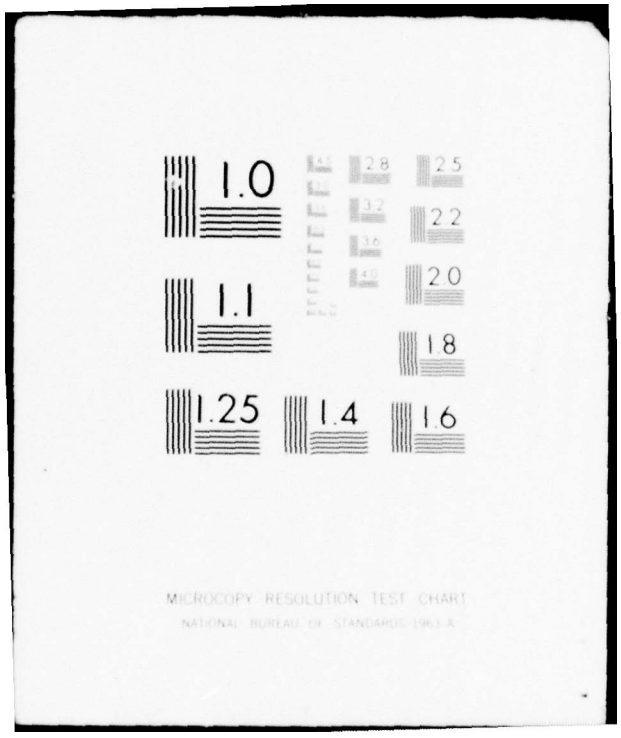
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MAGNETIC LPE CRYSTALLINE FILMS FOR SMALL BUBBLE DIAMETER CYLINDRICAL-DOMAIN MEMORY APPLICATIONS

M. Kestigian, W. R. Bekebrede and A. B. Smith

Sperry Research Center
100 North Road
Sudbury, Massachusetts 01776

August 1977
Technical Report AFAL-TR-77-168
Interim Report for Period January – June 1977

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Dr. M. G. Mier

Signature
Name: MILLARD G. MIER
Project Engineer/Scientist

FOR THE COMMANDER

Robert D. Larson

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Rare-earth iron garnet crystalline compositions were formulated, films deposited by liquid-phase epitaxy, and magnetic evaluation measurements performed. The results of the magnetic property experiments were used to select improved rare-earth iron garnet compositions and film deposition conditions for small-bubble-diameter magnetic memory applications. Representative results of delivered samples are given in tabular form in the Appendix. During this report period, samarium thulium-, yttrium samarium thulium-, yttrium samarium lutetium-, and yttrium europium thulium gallium iron garnet films were grown and their static magnetic properties evaluated. Yttrium samarium lutetium calcium germanium iron garnet small-bubble-diameter films were prepared and evaluated for comparison of their magnetic properties. (Continued on reverse side)

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20. ABSTRACT

In addition to the static magnetic property experiments, dynamic measurements were performed on $(LaEuTm)_3$ $(FeGa)_5O_{12}$ and $(SmTm)_3$ $(FeGa)_5O_{12}$ films. Mobilities of 233 and 140 cm/sec/Oe, respectively, were determined for representative samples. These mobility figures reveal that these two small-bubble-diameter compositions would operate at a half megabit data rate.

PREFACE

This project was initiated by the Air Force Office of Scientific Research (AFSC), United States Air Force under the direction of Dr. Millard Mier (AFAL/DHE). This is the Interim Technical Report for the period January 1977 through June 1977. The first Interim Technical Report, for the period June – December 1976, was published as AFAL-TR-77-33, April 1977.

The research described was carried out at the Sperry Research Center, Sudbury, Massachusetts 01776. The project manager at SCRC is M. Kestigian.

Project Monitor: Dr. Millard Mier

Contract Number: F44620-76-C-0121

Contract Report Number: SCRC-CR-77-45

Reporting Period: January through June 1977

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M. Kestigian
A. B. Smith

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SECTION I

SUMMARY

Rare-earth iron garnet crystalline compositions were formulated, films deposited by liquid-phase epitaxy, and magnetic evaluation measurements performed. The results of the magnetic property experiments were used to select improved rare-earth iron garnet compositions and film deposition conditions for small-bubble-diameter magnetic memory applications. Representative results of delivered samples are given in tabular form in the Appendix. During this report period, samarium thulium-, yttrium samarium thulium-, yttrium samarium lutetium-, and yttrium europium thulium gallium iron garnet films were grown and their static magnetic properties evaluated. Yttrium samarium lutetium calcium germanium iron garnet small-bubble-diameter films were prepared and evaluated for comparison of their magnetic properties. In addition to the static magnetic property experiments, dynamic measurements were performed on $(LaEuTm)_3\ (FeGa)_5O_{12}$ and $(SmTm)_3\ (FeGa)_5O_{12}$ films. Mobilities of 233 cm/sec/Oe and 140 cm/sec/Oe, respectively, were determined for representative samples. These mobility figures reveal that these two small-bubble-diameter compositions would operate at a half megabit data rate.

SECTION II

OBJECTIVES

This research is concerned with the preparation, characterization and evaluation of crystalline garnet magnetic films. The liquid-phase epitaxial growth technique was used to deposit magnetic thin films on commercial non-magnetic 3G substrates. These thin films were evaluated for use in small bubble diameter cylindrical domain memory devices. Research performed in addition to formulation and thin film deposition studies included measurement of wall energy, anisotropy, temperature coefficient, temperature range and magnetization. Analyses involved the presence of impurities, nonstoichiometry and charge compensation considerations. The goal is to prepare and evaluate a small bubble diameter (less than 2 μm) LPE crystalline garnet film with the following characteristics:

wall energy density = 0.25 ergs/cm^2

$q = H_k/4\pi M_s > 3$

velocity > 1000 cm/sec @ ΔH = 5 Oe

temperature coefficient = 0.2%/^{o}C

rotating field drive at 10^6 bit/sec shift rate < 25 Oe

SECTION III

SPERRY RESEARCH CENTER TECHNICAL APPROACH

The overall technical approach to be used in the development of a small-bubble-diameter cylindrical-domain mass-memory material emphasizes the formulation, preparation, characterization, evaluation and testing of magnetic crystalline thin films.

The rare earth iron garnet magnetic thin films have been found to be the most promising 3 to 8 μm bubble diameter materials for bubble memory devices. Large cross sectional area films of suitable perfection and desirable magnetic properties have been obtained from liquid-phase epitaxial deposition experiments. It is reasonable to assume that these successes can be extended to include small-bubble-diameter garnet compositions.

Gadolinium gallium garnet (3G) has found widespread use as the nonmagnetic substrate material for the LPE deposition of magnetic garnet thin films. No doubt research extended to include 1 to 2 μm bubble diameter materials will also utilize 3G substrates. Since polished 3G substrate slices of adequate quality are readily available commercially, gadolinium gallium garnet boules will not be grown. However, if for any reason commercial substrate sources are not adequate for the deposition of small-bubble-diameter thin films, nonmagnetic garnet single crystals will be grown from the direct melt by the Czochralski technique, oriented crystallographically, cut, polished and cleaned prior to use.

The approach to be followed in the growth of magnetic crystalline films will be the liquid-phase epitaxial method. This technique has proven

to be the superior method for obtaining high perfection magnetic films. While both tipping and dipping modifications of LPE growth have been employed, the horizontal-wafer-dipping reverse rotation process will be used for the growth of small-bubble-diameter crystalline thin films.

The selection of the optimum small bubble diameter crystalline garnet composition will take into consideration the results of several fundamental magnetic property measurements. Dynamic conversion, hard bubble suppression, propagation angle, mobility, coercivity, temperature dependence of magnetic properties, and anisotropy are parameters that must be investigated and understood. These experiments must be supplemented by magnetization, bubble diameter and bubble collapse measurements on all samples.

Compositions to be grown and evaluated include samarium thulium-yttrium samarium lutetium- and europium thulium iron garnet all of which contain gallium as the non-magnetic cation diluent, and yttrium samarium lutetium calcium- and yttrium europium lutetium calcium iron garnet which contain germanium as the non-magnetic cation. Investigations will be conducted with the objective of preparing a rare earth iron garnet composition which concentrates all of the transition metal nonmagnetic cations exclusively in the tetrahedral site. The use of germanium instead of gallium approaches this condition. Another approach that might prove to be superior would be to use vanadium, together with a monovalent cation for charge and cation compensation.

SECTION IV

SUBSTRATE PREPARATION AND CHARACTERIZATION

Gadolinium gallium garnet (3G) substrates have been obtained as polished wafers from Allied Chemical Company. The specifications under which these wafers were purchased are as follows:

Diameter = 1 inch

Thickness = 0.020 inch

Flat to 3 fringes over central 85% of area

Core, birefringence, and inclusion free

Crystallographically oriented to within 0.5 degree of $\{111\}$.

Five or fewer defects over central 85% of area, as revealed by a 2-minute etch in 220° C phosphoric acid, using Nomarski interference contrast microscope.

Each 3G wafer, immediately prior to being used as an epitaxial substrate, is cleaned by the following sequence of steps:

1. Rinse in acetone
2. Rinse in demineralized water
3. Boil in trichloroethylene for $\frac{1}{2}$ hour
4. Boil in 10% sodium hydroxide for $\frac{1}{2}$ hour
5. Rinse in demineralized water
6. Immerse in phosphoric acid at 120°C for 1 minute
7. Rinse in hot tap water
8. Rinse in demineralized water
9. Blow dry with filtered air gun

SECTION V

LIQUID-PHASE EPITAXIAL FILM DEPOSITION

The basic liquid-phase epitaxy (LPE) growth procedure used throughout this contract period is conventional for bubble memory films and utilizes horizontal dipping of [111] crystallographically oriented $Gd_3Ga_5O_{12}$ (3G) polished substrates.

The substrate is cleaned prior to use and is supported by a three-pronged platinum wire holder. A lowering-rotation mechanism is used to position the substrate above the solution for pre-heat purposes until temperature equilibrium is reached. Excessive exposure to the vapors above the solution causes defects to form, whereas insufficient heating results in uncontrolled film deposition. The growth process must be carried out under isothermal conditions. Any temperature fluctuations during the growth process produce pronounced film property differences.

Kanthal wound-electrically heated-resistance furnaces were used in the LPE experiments. The temperature profile in a single zone furnance is determined largely by furnace geometry, conduction losses from the furnace ends and by the position of baffles which minimize convection currents. A zone uniform in temperature to ± 1° was 8 cm in length and decreased by 2° one half inch above the solution surface.

Garnet films were grown on [111] 3G substates by LPE techniques previously described by numerous researchers. During this study, the

substrates were rotated-reverse rotated with a 2-second period at a rate of 60 rpm. Rotation rates less than 30 rpm and greater than 100 rpm led to a degradation of thickness uniformity. A 600 rpm rotation was used when the grown film was withdrawn from the solution. This procedure resulted in obtaining higher quality magnetic films, as any flux residue that had adhered to the film was removed quickly by this procedure.

Succeeding LPE film growth experiments were carried out after immediate magnetic property measurements were performed. These characterization studies included lattice-match-mismatch, film thickness, bubble diameter, magnetization, ℓ , q , and anisotropy measurements. Adjustment in solution composition, deposition procedure and deposition conditions were made on the basis of these evaluation measurements. We realize these evaluation studies do not include dynamic properties; however, unless a film composition exhibits the desired static magnetic properties, it will not meet contract objectives. What this preliminary evaluation procedure does accomplish is that sufficient results are obtained to direct suceeding film growth studies with a minimum of lapsed time.

Saturation temperature T_S was defined for each solution as the temperature at which the growth rate was just discernible (less than 0.05μm/min for a minimum growth time of 10 minutes. Film deposition was carried out 10 to 20° below the observed saturation temperature for any given solution.

Distribution coefficients employed during this phase of the program were controlled such that the garnet phase was the stable species in any growth process, regardless of film deposition or solution composition

modifications. A listing of all R values and/or adjustments would serve no meaningful purpose and is omitted to conserve space and to yield a simpler, more managable report.

During the course of this contract, over 480 LPE film depositions have been made in the search for an improved small bubble diameter crystalline composition. A typical melt composition for the LPE growth of $(YSmTm)_3 (GaFe)_5O_{12}$ garnet films in mole per cent is as follows:

Y_2O_3	0.149
Sm_2O_3	0.057
Tm_2O_3	0.028
Ga_2O_3	0.544
Fe_2O_3	8.915
PbO	85.187
B_2O_7	5.120

This preparation yields films with less than two micrometer bubble diameter at a growth rate of approximately 1.0 μm/minute.

SECTION VI

MAGNETIC FILM EVALUATION TECHNIQUES

The techniques for measuring static bubble parameters have been described in the first interim report. Since that report was written, we have also measured the bubble velocity vs. drive field characteristics of several promising small-bubble materials. These measurements have been made using the bubble-shift technique originally described by Vella-Coleiro and Tabor[1]. In this technique, two parallel conductors are placed against the surface of the garnet as shown in Fig. 1. To make the measurement, a bubble is positioned midway between the conductors and the conductors are pulsed. The initial and final positions of the bubble are measured using a polarizing microscope and from this displacement and the pulse length, the velocity can be calculated.

In utilizing the bubble-shift technique, we realize that bubble behavior under dynamic conditions is too complex to be fully characterized by one measurement. It has been reported[2], for example, that a significant amount of the bubble motion may actually take place after the drive pulse has ended. Also, dynamic conversion effects[3] can occur causing the apparent bubble mobility to depend on the amplitude of the driving field. In spite of these complications, we believe that the bubble-shift technique is adequate for our present purposes. Because it does measure the movement of actual bubbles and because a full range of drive fields can readily be applied, this technique should provide a valid initial comparison between potential bubble materials.

Although many bubble-shift velocity measurements are reported in the

literature, almost none of these data are for bubbles of 3 μm diameter or smaller because of the experimental difficulties in making such measurements. Even when using the best polarizing microscopes, the measurement of bubbles of this size is made virtually impossible by the small dimensions involved and the poor contrast of the image. In order to make such measurements possible, we have purchased a special television system made to our specifications. This system utilizes an ultra-low-light-level camera tube having an integral silicon image intensifier. The output of this tube is then electronically processed to enhance the image by introducing controllable amounts of contrast enhancement, clipping, and high-frequency peaking. In addition, a pair of measuring lines is added electronically to the image. The position of these lines is fully adjustable and the distance between them is displayed on a digital meter. A block diagram of this system is shown in Fig. 2 and a photograph of the apparatus is shown in Fig. 3. We find that this apparatus does allow us to make the small-bubble measurements that previously were either difficult or impossible. Velocity measurements can now be made on bubbles down to 1 μm in diameter. In addition to these dynamic measurements, all the static measurements (and particularly the strip-width measurement) are made much more conveniently and accurately with the use of the television system.

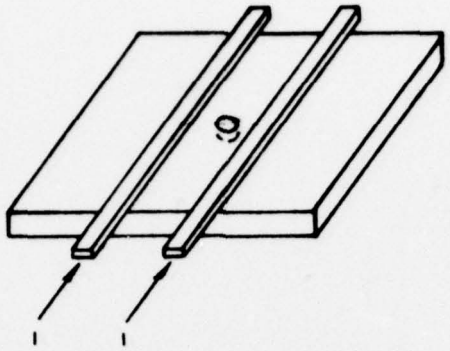

FIGURE 1 Sketch showing the basic configuration used for the bubble-shift measurement of velocity employing two conductors carrying a pulsed current I.

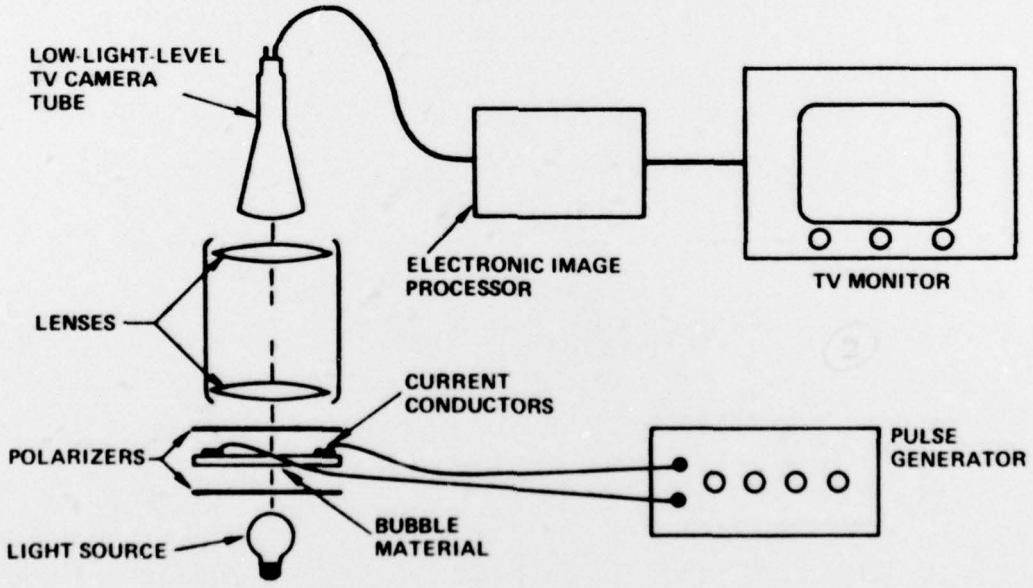

FIGURE 2 Block diagram of a television monitoring system used for measurements of small bubble materials.

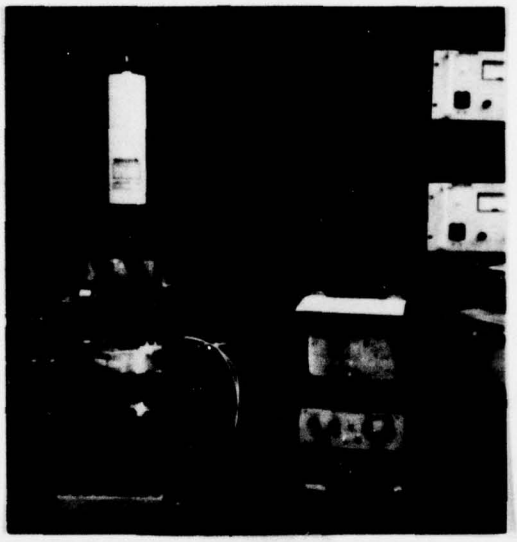

FIGURE 3 The microscope and television monitoring system used for the measurement of small bubble materials.

SECTION VII

MAGNETIC FILM PROPERTY MEASUREMENTS

In the first interim report, we identified two promising materials for small bubble devices on the basis of their static properties. These materials were $(SmTm)_3\ (GaFe)_5O_{12}$ and $(LaEuTm)_3(GaFe)_5O_{12}$. In addition to the room temperature values of all the important bubble parameters, the temperature dependence of stripe width and collapse field for these materials were also given in that report. In order to fully assess the usefulness of these materials for device use, it is also necessary to examine the variation of the anisotropy constant K_u with temperature. If the anisotropy field $H_k = \frac{2K_u}{M}$ falls below some minimum value, spontaneous nucleation of bubbles can occur in device use. The minimum anisotropy that can be tolerated varies with the circuit type, but one rule of thumb that is frequently used is that the $q = \frac{H_k}{4\pi M}$ should be greater than 3. The results of anisotropy measurements of these two materials are presented in Figs. 4 and 5 where we have plotted the data three different ways, i.e. in terms of K_u, H_k, and q.

We see from Fig. 4 that the q of $(SmTm)_3\ (GaFe)_5O_{12}$ is 5.2 at 125°C. Since this sample has an ℓ of 0.16 μm, it would support bubbles with a diameter of approximately 1.6 μm. Since q scales with ℓ, as described in the first interim report, this material could be adjusted to provide 1 μm bubbles with a $q > 3$ at 125°C. This result together with the data presented in the first interim report all indicate that this material is well suited

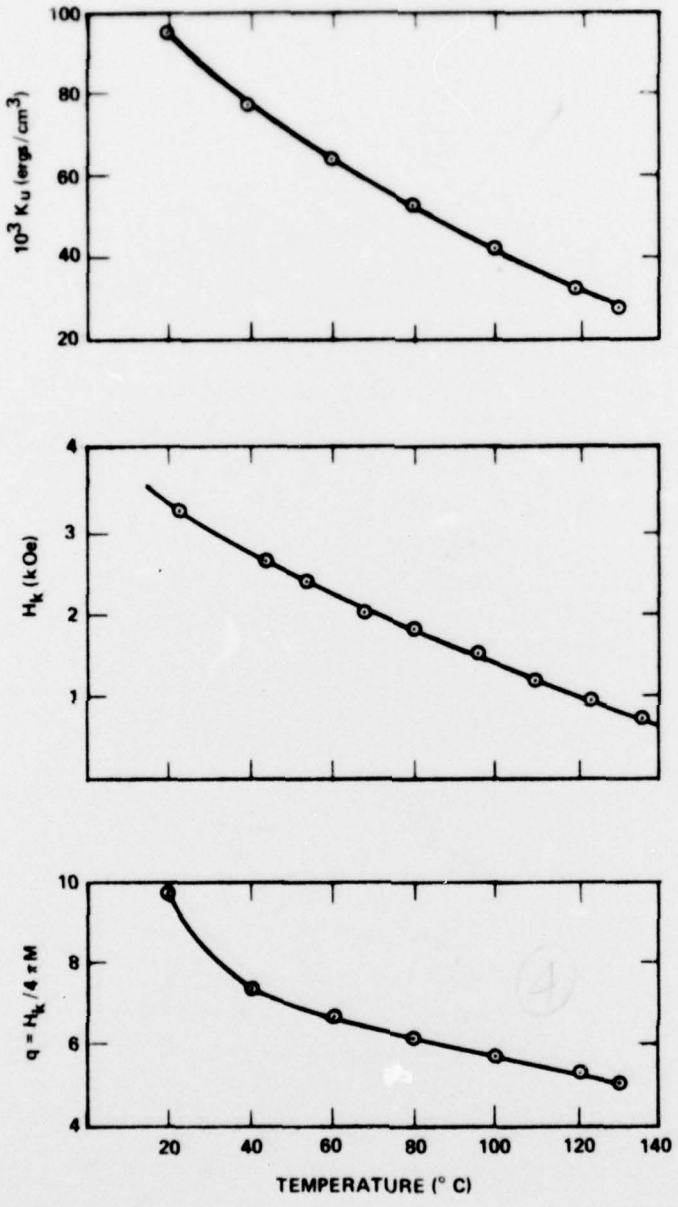

FIGURE 4 K_u, H_k and q vs. temperautre for a $(SmTm)_3(GaFe)_5O_{12}$ film. (The magnetic parameters of this film at room temperature are: $\ell = 0.16\ \mu m$ $4\pi M = 645$.G.)

for bubble devices using bubbles as small as 1 μm and covering the entire military temperature range of -55° C to +125° C. Similarly, the data in Fig. 5 and that in the first interim report indicate that $(LaEuTm)_3(GaFe)_5O_{12}$ will provide $q > 3$ for 1.5 μm bubbles and also operate over the full military range.

Having established that the static bubble properties of both $(SmTm)_3(FeGa)_5O_{12}$ and $(LaEuTm)_3(GaFe)_5O_{12}$ are favorable for small bubble applications, we must also determine whether their dynamic behavior is acceptable. Therefore we have measured the bubble-shift velocity v vs. drive field ΔH for both of these materials. The results are shown in Figs. 6 and 7 where we have also indicated for each material the mobility determined[1] from the slope of the curve.

We see from Fig. 6 that $(SmTm)_3(FeGa)_5O_{12}$ has a mobility of 140 cm/sec/Oe. This value is considerably lower than those commonly obtained with 6μm-diameter bubble materials. However, a smaller mobility can be tolerated in small-bubble devices since the bubble does not have to move as far between circuit positions. For example, let us assume a bubble diameter of 1 μm. The circuit period will then be about 4 μm. Assuming a ΔH of 10 Oe, Fig. 6 shows that the velocity will be 480 cm/sec which is 1.2 periods per microsecond. The theoretical maximum data rate would therefore be 1.2 megabits per second. However, since the velocity is not constant as the bubble transverses a practical circuit, the maximum actual data rate would probably be reduced by a factor of roughly three, yielding a rate of ~ 400 k bit/sec.

Thus according to this estimate, $(SmTm)_3(GaFe)_5O_{12}$ has reasonable device speed capabilities but it does not meet the ideal specifications set

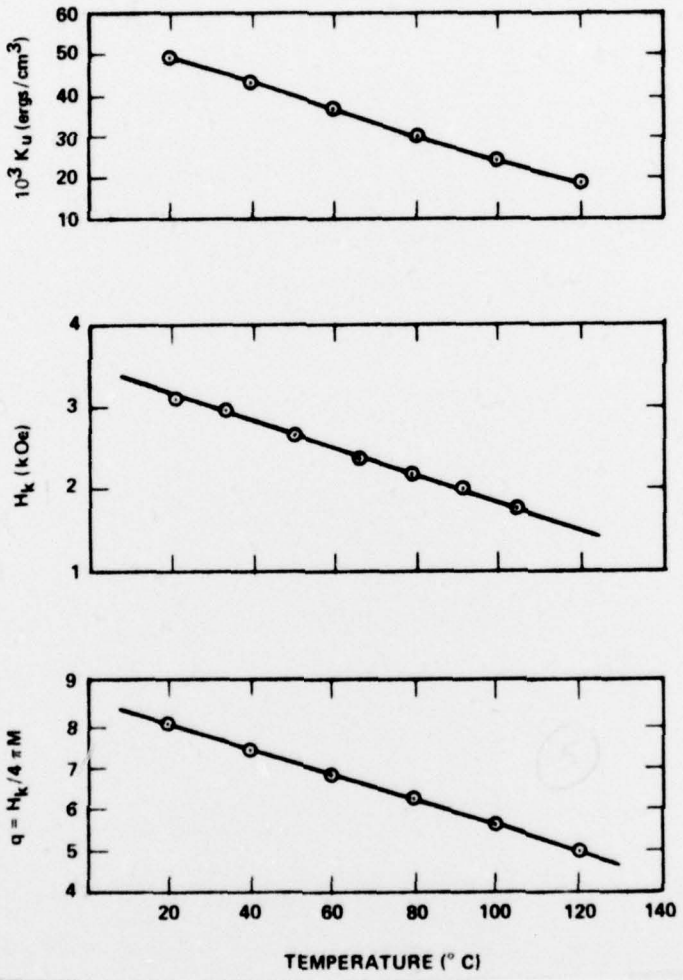

FIGURE 5 K_u, H_k and q vs. temperature for $(LaEuTm)_3(GaFe)_5O_{12}$. (The magnetic parameters of this film at room temperature are: $\ell = 0.24\ \mu m$, $4\pi M = 533.G.$)

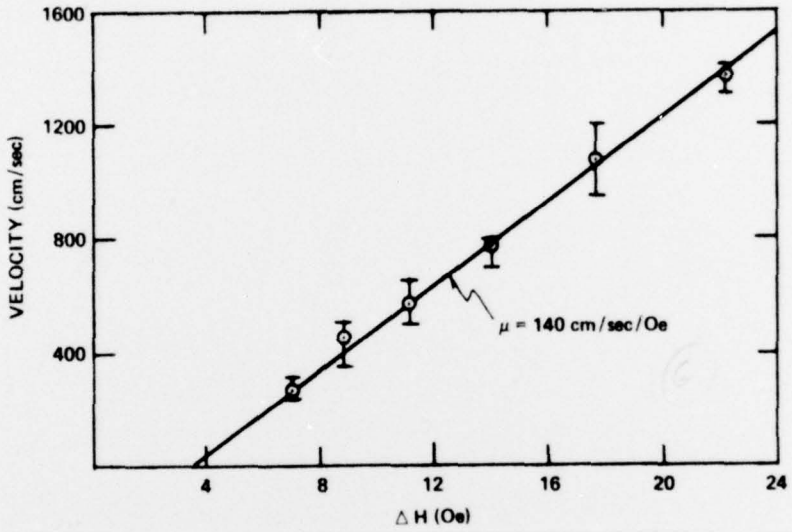

FIGURE 6 Velocity vs. drive field for a $(SmTm)_3(FeGa)_5O$ film having the following properties: thickness = 3.6 μm, ℓ = 0.16 μm, 4 π M = 645G., H_k = 4657.Oe, $K_u = 1.2 \times 10^5$ ergs/cm^3, q = 7.4. (The bars indicate the spread in the data obtained upon repeating each measurement about six times; the circles represent the average of these six measurements. The spread indicated by the bars is due primarily to variability in bubble behavior, not to uncertainties in the measurement.)

forth in section I. As shown by Fig. 7, $(LaEuTm)_3\ (GaFe)_5O_{12}$ has a considerably higher mobility and therefore would be capable of a correspondingly higher device speed. Its anisotropy is about half as big, however, so its minimum bubble size for q = 3 will be $\sim$ 1.5 μm. In comparing these two materials we see that, there is a trade off between high anisotropy and high mobility i.e. the material with a higher value of one of these parameters has a lower value of the other. A further example of such a trade off is given by the data in Fig. 8 on $(YSmLuCa)_3\ (FeGe)_5O_{12}$. This material has a mobility of 1000 cm/sec/Oe but a much lower anisotropy so that the minimum bubble size for q = 3 at room temperature is about 3. μm. The ideal bubble material would, of course, have both high mobility and high anisotropy. The materials described above represent the best combination of properties for small-bubble applications yet discovered, but further work will be required to satisfy the contract goals stated in Section I.

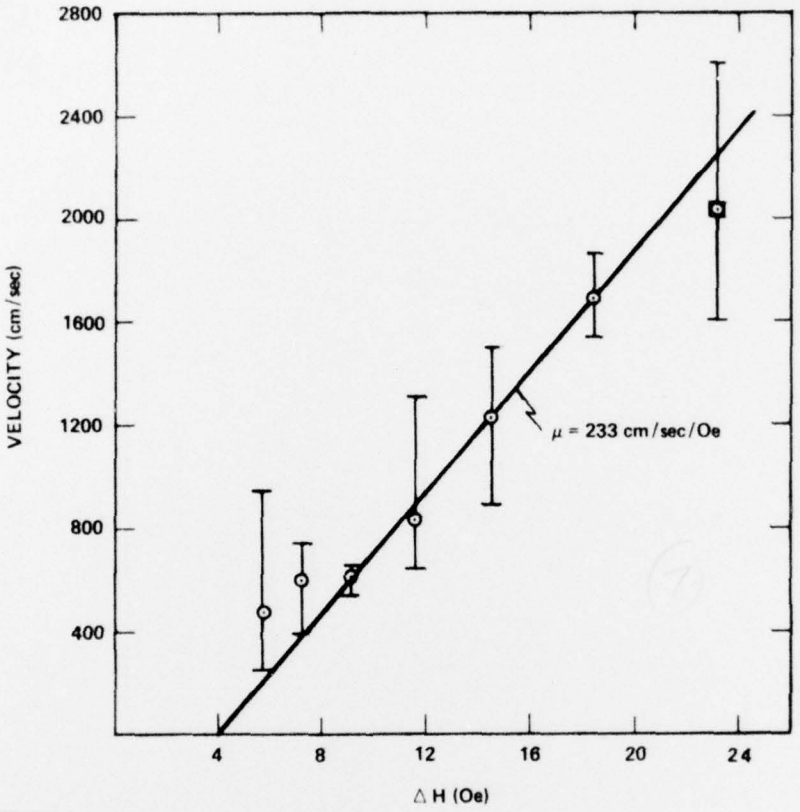

FIGURE 7 Velocity vs. drive field for a $(LaEuTm)_3(GaFe)_5O_{12}$ film having the following properties: thickness = 3.6 μm, ℓ = 0.24, 4 π M = 480, H_k = 3276.Oe, $K_u = 6.3 \times 10^4$ ergs/cm^3, q = 6.9. (The bars indicate the spread in the data obtained upon repeating each measurement about six times; the circles represent the average of these six measurements. The spread indicated by the bars is due primarily to variability in bubble behavior, not to uncertainties in the measurement.)

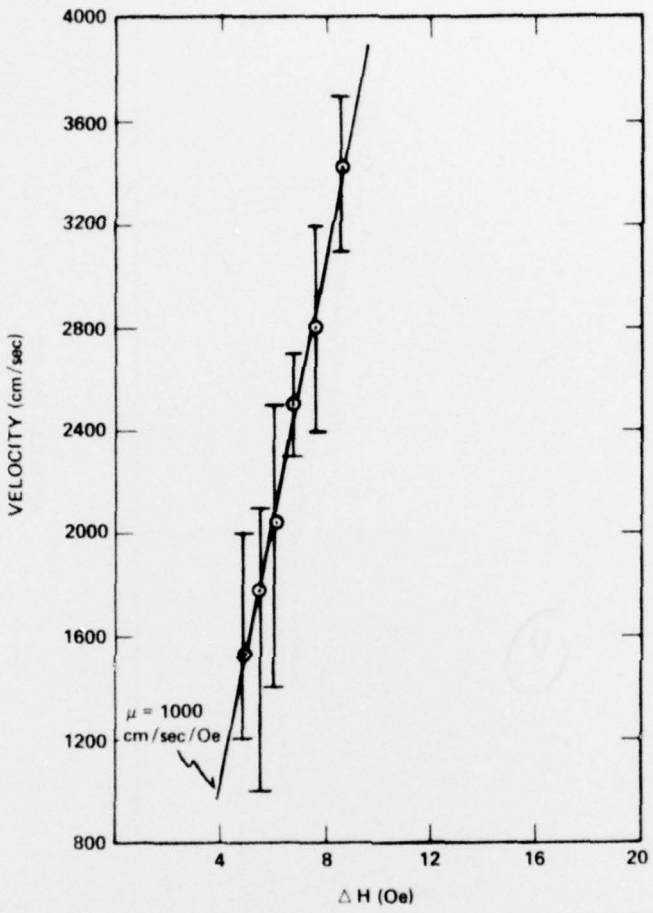

FIGURE 8 Velocity vs. drive field for a $(SmTm)_3(FeGa)_5O_{12}$ film having the following properties: thickness = 2.3 μm, 4 π M = 320.G, H_k = 670.Oe, $K_u = 9.7 \times 10^3$ ergs/cm^3, q = 2.4. (The bars indicate the spread in the data obtained upon repeating each measurement about six times; the circles represent the average of these six measurements. The spread indicated by the bars is due primarily to variability in bubble behavior, not to uncertainties in the measurement.)

APPENDIX

TABULATION OF MAGNETIC DATA FOR SAMPLES DELIVERED TO CONTRACT MONITOR

Date of Shipment to WPAFB	SAMPLE NUMBER	SAMPLE COMPOSITION	FILM THICKNESS (μm)	ZERO-FIELD STRIPE WIDTH (μm)	$4\pi M$ (G)	ℓ (μm)	$10^{-4} K_u$ (ergs/cm^3)	q
1/26/77	1	$(SmTm)_3(FeGa)_5O_{12}$	2.7	1.8	-	-	-	-
	2	"	2.7	1.8	-	not measured	-	-
	3	"	2.7	1.8	-	-	-	-
	4	"	2.8	1.8	-	-	-	-
	5	<111> G^3 polished substrate						
2/18/77	1	$(YSmLu)_3(FeGa)_5O_{12}$	2.0	1.5	-	-	-	-
	2	"	2.5	1.4	-	-	-	-
	3	"	3.0	1.4	-	-	-	-
3/24/77	1	$(YSmLu)_3(FeGa)_5O_{12}$	3.1	1.8	559	0.138	2.85	2.3
	2	"	3.1	1.5	555	0.098	3.67	3.0
	3	"	3.1	1.5	548	0.098	3.76	3.2
	4	"	3.9	2.1	626	0.150	5.34	3.4
	5	<111> G^3 polished substrate						
4/21/77	1	$(YEuTm)_3(FeGa)_5O_{12}$	2.9	1.5	-	-	-	-
	2	"	2.7	1.5	-	-	-	-
	3	"	3.0	1.5	-	-	-	-
	4	"	2.9	1.5	-	-	-	-
	5	<111> G^3 polished substrate						
5/18/77	1	$(YSmLu)_3(FeGa)_5O_{12}$	2.0	1.2	-	-	-	-
	2	"	2.1	1.25	-	-	-	-
	3	"	2.3	1.35	-	-	-	-
	4	"	2.0	1.3	-	-	-	-
6/30/77	1	$(YSmTm)_3(FeGa)_5O_{12}$	2.4	1.8	699	0.169	11.45	6.2
	2	"	3.3	2.0	832	0.160	10.7	3.9
	3	"	4.3	1.7	759	0.096	4.5	2.0
	4	"	3.5	1.75	762	0.117	6.3	2.7

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